Effects of a Chitosan Coating Layer on the Surface Properties and Barrier Properties of Kraft Paper

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Biodegradable chitosan can be applied as a coating on the surface of kraft paper in order to improve its barrier properties against water vapor and air. The food packaging industry can benefit from the addition of chitosan to its current packaging, and in turn reduce pollution from plastic packaging plants. This paper discusses the film formation of chitosan, the permeability of paper coated with a chitosan layer, and the influence on the paper's surface and barrier properties under different process conditions. SEM (scanning electron microscope), AFM (atomic force microscope), ATR-FTIR (Fourier transmission infrared spectroscope with attenuated total reflection), and PDA (penetration dynamics analysis) were used to analyze the properties of chitosan's film formation and permeability. A controlled experiment showed that the chitosan layer was smoother than the surface of the uncoated kraft paper, had better film formation, and that there was no chitosan penetration through the kraft paper. The barrier properties against water vapor were strongest when there was a higher concentration of chitosan solution at the optimum pH, stirring speed, and those with a thicker coating on the kraft paper.

Keywords: Chitosan; Film formation; Permeability; Barrier properties

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INTRODUCTION

Although plastic is widely used in the packaging industry, a negative characteristic of the material is that it easily causes pollution to the environment due to it being non-biodegradable. Thus, newer packaging materials, which are biodegradable, green, nontoxic, as well as possessing better barrier properties to oxygen and carbon dioxide, have commanded increasing attention. Considered a "green" package material, paper has been increasing every year, but with its poor barriers against oxygen and water vapor, its application in the food packaging industry has faced certain limitations.

Chitosan is a naturally renewable resource, and it is second to cellulose in available quantity as a natural polysaccharide (Liu and Chen 2011). It is a biodegradable polymer, having the composition 2-amino-2-deoxidation-beta-D glucose connected through a β-(1-4) glycosidic bond, and its structure is very close to that of cellulose. Generally, chitin is easily extracted from the shells of prawns, crabs, insects, or fungal mycelia (Qin and Guo 2007), and chitosan can be obtained through the deacetylation reaction of chitin. Chitosan has been widely applied because it is biodegradable, biocompatible, and it has high microbial resistance. Food packaging film created with chitosan would have antibacterial

properties, and it could improve food safety, as well as prolong the shelf life of food (Muzzarelli and Muzzarelli 2005).

Chitosan can improve the barrier properties and expand the application range of traditional paper, especially in the food packaging industry. Vimaladevi *et al.* (2015) studied the packaging performance of chitosan film in combination with the use of organic acids. Mohammed (2010) discussed the production of chitosan composite biological film, based on the antibacterial activity and biodegradability of chitosan, and they discussed its potential value in the food storage and packaging industry. Lambertus *et al.* (2014) discussed the latest developments of chitosan film. Chitosan has the ability to improve the internal environment of packaging food by producing a semi-permeable condition and reducing the transpiration rate (Takahashi *et al*. 2007). In addition, Mukoma and coauthors (2004) discussed the methanol permeability of chitosan by means of preparing chitosan film. Casariego *et al.* (2009) studied a nano-composite packaging film, which was prepared from using dispersed clay micro-nano particles filled with chitosan, in order to enhance the barrier properties of paper.

Chitosan can be easily coated onto the surface of paper or a board, which is one of the most effective approaches to improving the barrier properties of paper, or board, to oxygen and water vapor. The key factors that influence the barrier properties of chitosan film are the formation and permeability of the film on the surface of paper. The present research used kraft paper as a base, and coated chitosan on the surface of the paper. Then the surface properties of the coated paper were analyzed and the effect on the water vapor barrier properties were evaluated for different chitosan concentrations. The dosage ratio of chitosan to glacial acetic acid and chitosan solution stirring speed were varied. The amount coated was calculated. The coated specimens were evaluated by use of a scanning electron microscope (SEM), atomic force microscope (AFM), Fourier transmission infrared spectroscope with attenuated total reflection (ATR-FTIR), and penetration dynamics analysis (PDA) detection methods. The formation and permeability of the coated paper were analyzed.

EXPERIMENTAL

Material

The base kraft paper had a basis weight of 120 g/m^2 (smoothness: 18.5 s with Bekk smoothness tester, elongation: 1.51% with Frank tensile testing machine). Chitosan was purchased from Guoyao Chemical Co., Ltd., China, with a degree of acetylation of 80% to 95%. The acetic acid, which had an assay of up to 99%, was purchased from Nanjing Chemical Industry Group (China).

Methods

The preparation of the chitosan solution

Chitosan powder was dispersed in distilled water with different concentrations of 1.0 wt.%, 1.5 wt.%, and 2.0 wt.%. The stirring speed was, respectively, 300 rpm, 500 rpm, 800 rpm, and 1200 rpm to the 1.5 wt.% chitosan solution. The effects of dosage ratio of glacial acetic acid to chitosan (dry weight)-30%, 50%, and 70% were studied. After stirring

for 1 h, the chitosan solution was purified by a 200-mesh sieve filter, then kept still for 30 min to allow elimination of foaming.

Coating

The term coater here refers to equipment used for coating some specific glues, coating materials, or printing inks on the surface of paper, then rolling the coated paper after drying. The coating weight was controlled by a roller coater (PK Print Coat Instruments Ltd., Litlington, Royston, Herts, SG8 0QZ, U.K.) having a model designation of K303 MULTI COATER. Through recording the additional weight of the kraft paper with the area of 210×297 mm after being coated and dried at 105 °C, the coating weights were obtained. The results of calculation were obtained for three valid digits.

Calendering

Calendering is often carried out on coated paper to compact the coating structure and to develop a greater level of smoothness. This experiment used the Model 300 mm calender apparatus, from the Nanjing institute of Light Industry. The operation was performed twice on the top side of the coated paper at 1 MPa pressure.

Scanning electron microscope (SEM) analysis

The patterns were cut into appropriate size and then stuck onto the sample stage with conductive double-sided adhesive tape. Then an ion sputtering apparatus was used for gold-plating the surface of the sample, and then the surface of the sample paper was imaged with the FEI Quanta 200 environmental scanning electron microscopy (SEM) at 15 kV acceleration voltage.

Atomic force microscope (AFM) analysis

Atomic force microscope analysis, using an AFM device of Bruker companies in the United States, was applied to study the film formation properties of the coating layer. The analysis adopted the probe tapping mode in 5×5 microns, on the surface of a pattern in air of a square range scanned and photographed. Using the supporting software, NanoScope Analysis 1.40, the corresponding patterns were drawn in a 2-dimensional and a 3-dimensional picture, and the RMS roughness of the surface was determined.

Attenuated total reflection spectroscopy (ATR-FTIR) analysis

ATR-FTIR was used to obtain organic compositional information of the sample by the reflection signal on the surface of the sample (Huang and Ji 2011). By measuring the uncoated and the chitosan coated paper top and bottom sides ATR-FTIR spectra, as well as analyzing the differences in their ATR-FTIR spectra, tests were carried out to determine whether the chitosan coating layer will penetrate from the top to the bottom side of the kraft paper.

The paper surface properties analysis

The smoothness and elongation properties of coated paper were tested according to China national standards *GB/450-455/1989*.

Penetration dynamics analysis (PDA)

A PDA curve was applied to analyze the water penetration and water absorption performance of the kraft paper coated with chitosan by the Penetration Dynamic analyzer (Liu and Chen 2011).

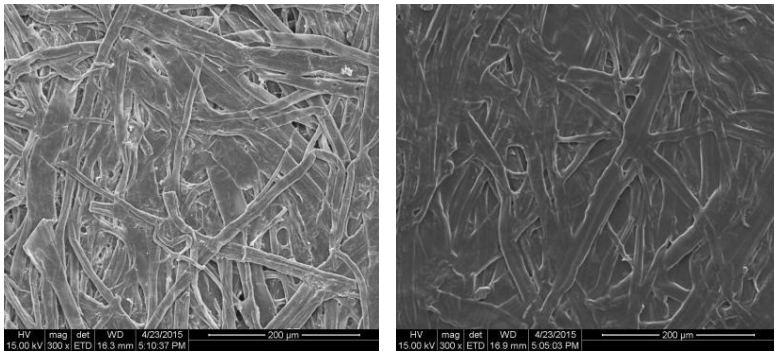
Water vapor permeability (WVP) analysis

Water vapor permeability was measured using the W3/060 WVP device. The temperature was 35 ± 2 °C with a humidity of 70% ± 2 °C. The test determined the extent to which water vapor at a certain temperature being drawn through the sample led to changes in the wet weight of the paper, measured in per unit area per hour, g/(m^2day). The penetrating direction of water vapor was perpendicular to the specific surface of the sample, and every surface of the sample would be maintained at a specific temperature and humidity (Wang and Zhao 2008).

RESULTS AND DISCUSSION

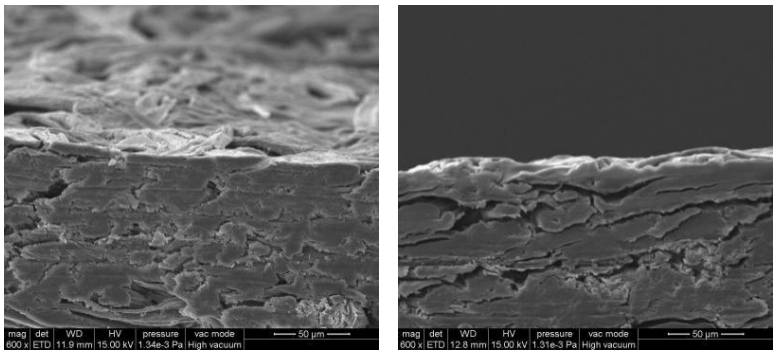
Effects of the Chitosan Film on the Surface of the Kraft Paper

The surface and cross section SEM images of the uncoated paper and kraft paper coated with 1.5 wt.% chitosan are shown in Figs. 1 and 2.



A B

Fig. 1. Chitosan-coated paper surface topography, (A) for the uncoated paper, 300×; (B) for 1.5 wt.% CS coated paper, 1.590 g/m^2, 300×

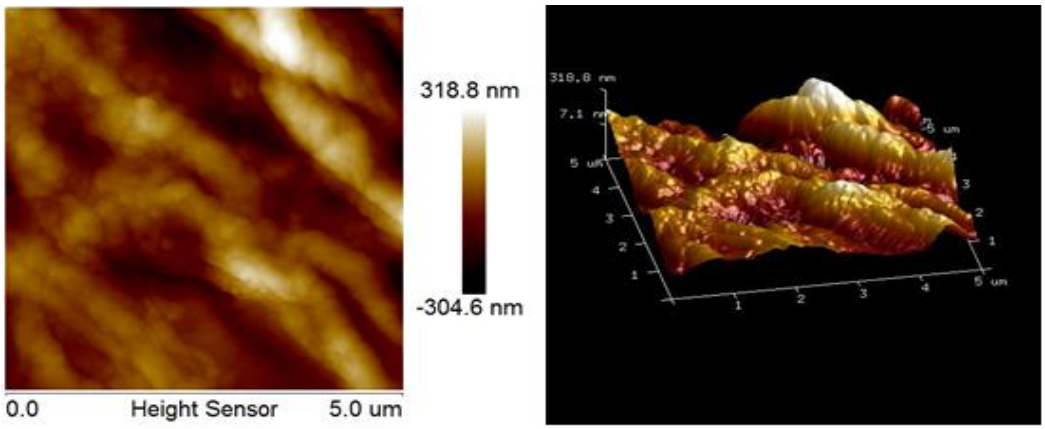


C D

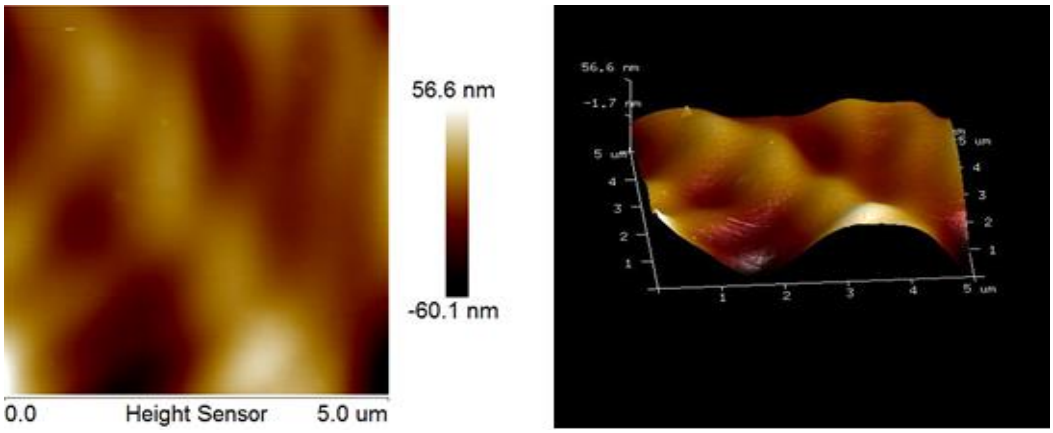
Fig. 2. Chitosan-coated paper cross-section topography; (C) for the uncoated paper, 600×; (D) for 1.5 wt.% CS coated paper，1.590 g/m^2，600×

It was apparent that the uncoated paper had a rough surface in which fibers crossed each other and formed lots of pores. In the process of coating chitosan on the uncoated paper, microporous pores were filled with chitosan, and a layer of chitosan film was formed on the surface of the uncoated paper (Dai and Long 2011). It is shown in Fig. 2 that a continuous chitosan film is formed and the paper's smoothness improved with the chitosan, compared with that of the uncoated one.

The surface of the uncoated paper and kraft paper coated with chitosan are shown in Fig. 3. A distinct difference was apparent in smoothness between the two different papers. There were numerous pores apparent on the surface of the uncoated paper, and it had a rough and uneven surface. The distance between the highest and the lowest point of the uncoated paper surface within the range of 5×5 micrometers was 623.4 nm, while the corresponding value for the chitosan-coated paper was 116.7 nm, indicating a reduction by 81.3%. This reduction was caused by the addition of chitosan-filled original pores on the paper surface, which formed a layer of film. Therefore, it resulted in changing the paper's surface morphology significantly, reducing the roughness in comparison to the uncoated kraft paper.



The uncoated paper



Chitosan coating weight of 1.590 g/m^2

Fig. 3. AFM images of the uncoated paper and chitosan-coated paper

The Root Mean Square (RMS) roughness of chitosan-coated paper is shown in Table 1. Compared to the uncoated paper, the RMS roughness of the chitosan-coated paper had decreased. When the chitosan coating weight was 1.590 g/m^2, the paper's RMS roughness was decreased by about 83.1%.

Table 1. Root Mean Square (RMS) Roughness of Chitosan-coated Paper

Type	Coating weight (g/m^2)	RMS roughness (μm)
The uncoated paper	0	0.0872
Chitosan-coated paper	1.590	0.0147

Based on these findings, chitosan was able to form a dense film on the surface of kraft paper and to improve the surface smoothness of the kraft paper greatly. The experiment demonstrated that chitosan had a good property of film formation and film uniformity on the surface of kraft paper.

Permeability Analysis of the Chitosan Film

ATR-FTIR was used to test whether or not the chitosan of the coating had permeated through the thickness of the kraft based paper in the thickness direction. Figures 4 and 5, respectively, show the ATR-FTIR spectra of the coated paper, of the top and bottom sides with chitosan.

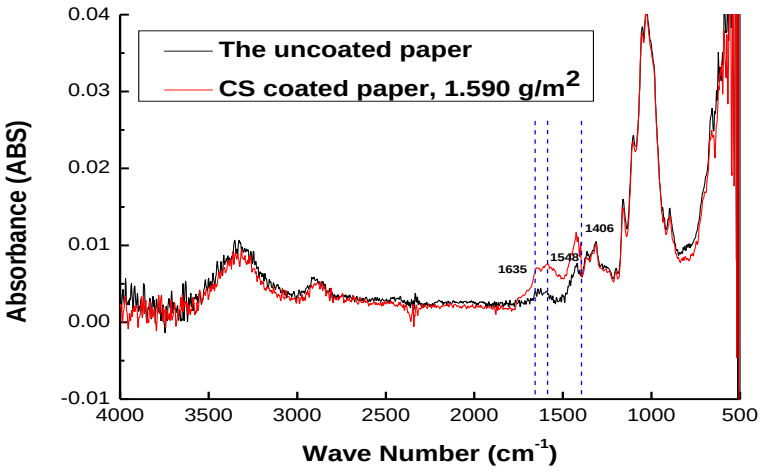


Fig. 4. Top side ATR-FTIR spectra of the uncoated paper and chitosan-coated paper

The ATR-FTIR spectra of the kraft paper showed absorption peaks of fibers, of which the main ingredient was cellulose. In fact, chitosan's structure is very close to that of cellulose. In the ATR-FTIR spectra, a peak in the range 3400 to 3200 cm^{-1} was attributed to the intermolecular hydrogen bonds in all the kraft papers' spectra. The absorption peaks at 2890 cm^{-1} and 1025 cm^{-1}, respectively, belong to the stretching vibration of C-H and C-O. From Fig. 4, a distinct C=O stretching vibration belonging to amide group or introduced by bonded water deformation at 1635 cm^{-1} was found in the top side spectra of chitosan-coated paper. The peak at 1548 cm^{-1} was assigned to the NH_3^+ bending vibration absorption peak, and 1406 cm^{-1} was assigned to stretching bands of C-N. These peaks were the characteristic absorption peaks of chitosan, although the intensity of the absorption peak

was not very strong. Based on Figs. 4 and 5, comparing the top and bottom sides spectra of the chitosan-coated paper, the characteristic absorption peaks of chitosan were not found in the bottom side spectra. These findings indicated that chitosan did not penetrate from the top side to the bottom side. In addition, there were the same spectral lines present on the bottom side between the uncoated paper and the chitosan coated paper.

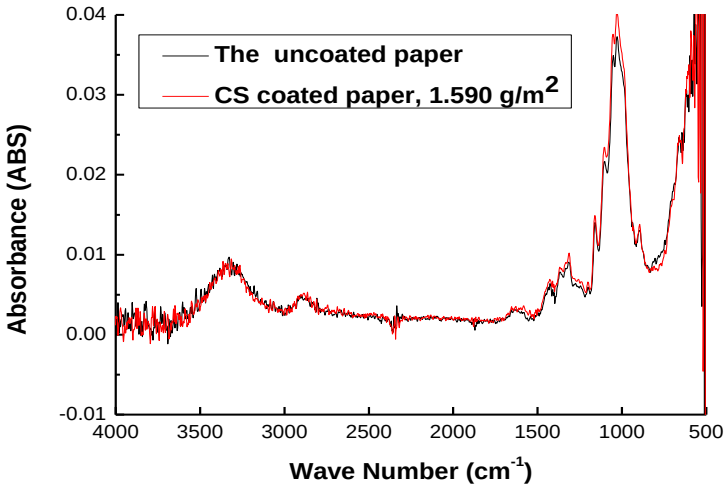


Fig. 5. Bottom side ATR-FTIR spectra of the uncoated paper and chitosan-coated paper

As shown, chitosan had a good film formation performance and remained on the top surface of the paper. It formed a layer on the paper surface, improving the smoothness and it decreased the roughness of the uncoated paper. In general, there was no permeation phenomenon happening when chitosan was coated onto the surface of the paper.

Effects of Different Process Conditions on the Properties of Coated Paper

The chitosan concentration, solution stirring speed, dosage ratio of chitosan (dry weight) to glacial acetic acid, and the coating weight are basic factors for the surface and physical properties of the coated paper. Design of experiments and results are shown in Table 2. All tests had the same coating weight.

Table 2. Results of the Coated Paper Surface and Physical Properties

NO.	Factors	Smoothness (s)	Elongation (%)
1	The uncoated paper	18.5	1.51
2	1.0 wt.%	22	1.74
3	1.5 wt.%	22.5	1.76
4	2.0 wt.%	23.5	1.79
5	300 rpm	28	1.83
6	500 rpm	29.5	1.83
7	800 rpm	34.5	1.94
8	1200 rpm	22	1.83
9	1:0.3	26	1.84
10	1:0.5	28	1.83
11	1:0.7	20	1.86

According to Table 2, with the concentration of chitosan solution increasing from 1.0 wt.% to 2.0 wt.%, there was little increase in the paper's smoothness. But compared with the uncoated paper, smoothness increased. With the increase of chitosan concentration, more chitosan molecules overlapped and intertwined with each other throughout a network structure, forming a dense chitosan layer on the surface of the paper (Chen *et al.* 2008). The chitosan itself has a certain strength, so the elongation of kraft paper coated with chitosan was bound to be enhanced. Thus, similarly, the elongation of chitosan coated paper was increased along with chitosan concentration.

It is clear from Table 2 that with the increase of the stirring speed, the smoothness of the coated paper increased gradually. But when the stirring velocity was 1200 rpm, the smoothness decreased. In this part, the concentration of chitosan solution was 1.5 wt.%. In fact, with the increase of stirring velocity, chitosan molecules would experience greater shear force and its molecular chains would progressively be broken. Scission of the chitosan macromolecule could decrease the viscosity of chitosan solution and improve the flow of it. But if the shear force was too intense, the film formation would be reduced. With the same theory, the elongation of the coated paper nearly presented the same tendency to changes of the smoothness.

When different dosages of acetic acid were added in chitosan solution, the pH of the chitosan solution would change accordingly. Results shown in Table 2 indicate that the smoothness of the coated paper was best when the dosage ratio of chitosan to acetic acid was 1:0.5. A relatively high pH value could reduce the degradation of chitosan (Liu and Chen 2011). With increasing dosage of acetic acid, fibers on the surface of the paper would undergo a quicker degradation process, which led to the destruction of the original fiber morphology of the uncoated paper, thus causing the film formation of chitosan to be worse and affecting the smoothness of the coated paper. However, the elongation of the coated paper exhibited little change with different pH. A higher concentration, and the optimum pH of the chitosan solution, as well as the optimum stirring speed, were helpful towards the film formation and smoothness of the coated paper. The effects of the coating weight on the smoothness and elongation of the kraft paper are shown in Fig. 6.

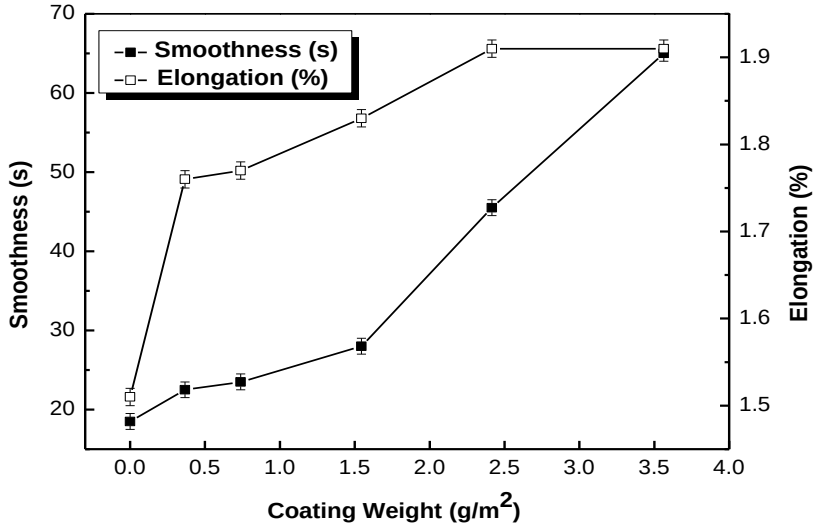


Fig. 6. Effects of chitosan coating weight on the coated paper

Based on the chitosan solution with the concentration of 1.5 wt.%, when the coating weight was 2.42 g/m^2, the smoothness of the kraft paper was increased by 146.0%, and elongation was increased by 26.5%, compared to the uncoated paper. The chitosan-coated paper with a higher coating weight possessed a thicker chitosan film between the chitosan and cellulose fibers, which were thicker and tighter. Therefore, the smoothness and elongation of the coated paper increased when the weight of the chitosan coating increased.

Effects of Different Process Conditions on PDA Curve of the Coated Paper

A PDA curve was applied to represent the penetration and absorption properties of the sample paper. The curve was obtained from the change in the crossing signal intensity of an ultrasonic wave with the contact time between water and the sample paper. The corresponding A60 for PDA curves are shown in Figs. 7 to 9, where A60 namely was the signal intensity of water crossing the pattern in 0 to 60 seconds. According to the picture, each curve exhibited a downward trend. The PDA curve of uncoated paper was steeper than any coated paper. The kraft paper had a poor surface compactness. When the paper specimens were immersed in the water, the fibers absorbed water directly, and their elastic modulus fell; thus the ultrasonic signal was falling fast. After the paper was coated with chitosan, a layer of chitosan film was present on the surface. The chitosan was infiltrated by water and began to absorb it, then the fibers began to swell. Due to the hydrogen bonds between the fibers being opened up, the paper strength began to weaken.

According to Fig. 7, along with the increase of the chitosan concentration, the crossing signal intensity at the same time presented an upward trend. It was the high concentration of chitosan-coated paper that easily formed a dense film and delayed water molecular movement from penetrating the paper.

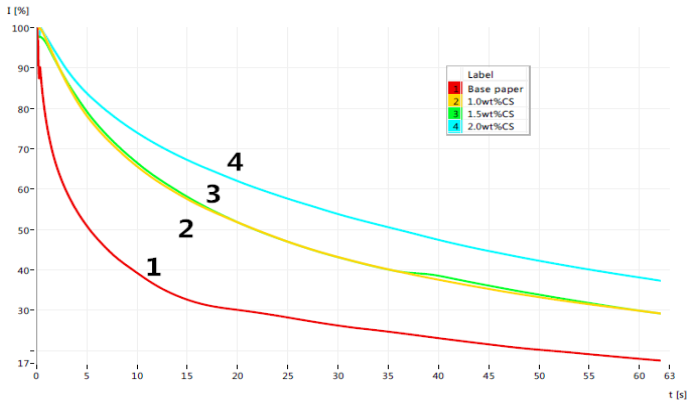


Fig. 7. Different concentrations of CS coated-paper PDA curve

Effects of the stirring speed of chitosan solution are shown in Fig. 8. Chitosan was dissolved in dilute acid, which was a gradual process. Stirring promoted dissolution of chitosan, but the dissolution also was accompanied by a small amount of degradation of chitosan (Jiang 2006). When the stirring speed was 800 rpm, chitosan-coated paper had the best effect on resisting water permeation. For this result, it showed that it was not easy for water molecular to penetrate chitosan-coated paper, when the chitosan solution had an optimal stirring speed at 800 rpm.

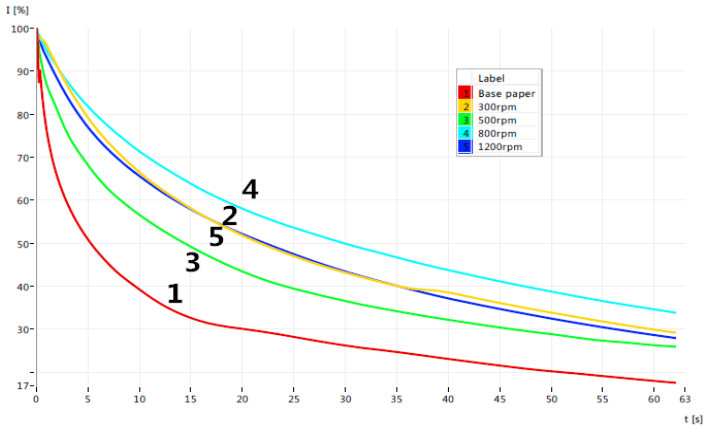


Fig. 8. Different dissolved stirring speeds of CS-coated paper PDA curve

The water permeability of chitosan solution with different acidity can be seen in Fig. 9. When the dosage ratio of chitosan to acetic acid was 1:0.5, the PDA curve of the corresponding coated paper was the slowest, and the crossing signal intensity was the highest over a certain period of time. The result indicated that there was a suitable pH for the chitosan solution to obtain a better influence on resisting to water permeation, more specifically, the optimum condition was that the dosage of acetic acid was one half of that of the chitosan (dry weight).

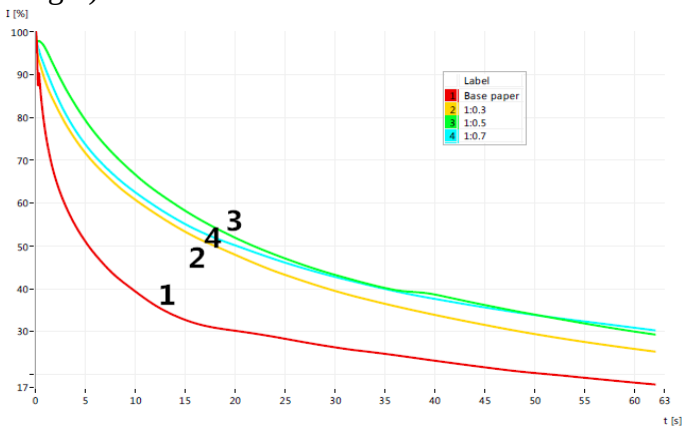


Fig. 9. Different amount of acetate CS coated paper PDA curve

In summary, the coated paper with a higher chitosan content had a better influence on resisting to water permeation. There was an optimum stirring speed of 800 rpm, and a dosage ratio of acetic acid to chitosan which was 1:0.5, for chitosan-coated paper to gain a better effect on resisting to water permeation. In addition, chitosan-coated paper with a higher coating weight presented a better performance of resisting water permeation.

Effects of Different Process Conditions on WVP of the Coated Paper

It is essential for packaging material to have excellent barrier properties. For example, food packaging paper must have good oxygen and water vapor barrier properties to make sure that the food will not become bad. The WVP test was applied to compare the

barrier properties for different materials. The chitosan film on the paper's surface had blocked the pores on the surface from fiber and pores formed by fiber crossing; thus it had an effect on barrier function.

As can be seen from Fig. 10, under the same coating weight, the coated paper had a low WVP when the chitosan had a high concentration. This was due to the presence of a higher concentration of chitosan, which resulted in closer connection between the chitosan molecules. Therefore, the chitosan film on the surface of paper were denser. As a result, it was difficult for water vapor molecules to penetrate through the coated paper.

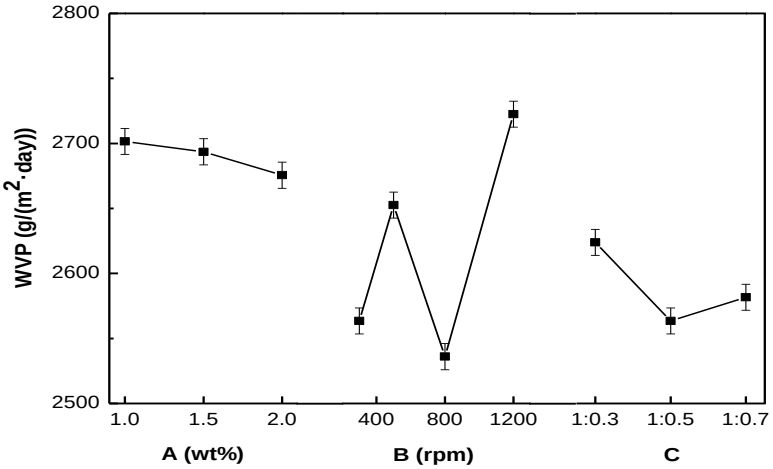


Fig. 10. The results of the coated paper WVP; (A) for chitosan concentration; (B) for stirring speed; (C) for the dosage ratio of chitosan to acetic acid. The WVP of the uncoated paper is 2808.24 (g/(m^2·day)).

In the process of preparing chitosan solution, stirring not only promoted the dissolution of chitosan, but the process also was accompanied by a small amount of degradation of chitosan. Once stirring too intense, it would make the chitosan chain degradation worse (Jiang 2006). From Fig. 10 (B), it was found the coated paper WVP declined after the first rise. Then it declined again with further increasing of the stirring speed. The chitosan-coated paper prepared with the stirring speed of 800 rpm had the lowest WVP. This trend might be attributed to the combined effect of the cleavage of chains and the entanglement of inter-chain in the process of gradual dissolution of chitosan. The optimal stirring speed at 800 rpm indicated the tortuosity of water vapor molecular permeable channel.

The pH of the chitosan solution also affected the WVP of the kraft paper with a chitosan coating. When the solution had an optimum acidity, namely, with a proper pH (the condition that the dosage ratio of chitosan to acetic acid was 1:0.5), the coated paper had a low WVP. In fact, in an alkaline condition, the fibers would swell, expand, and go soft, even though the chitosan exhibited less degradation. In such case, the amount of water vapor passing through the coated paper in a given time was increased.

The chitosan coating weight had a great effect on the WVP of a coated paper. This can be seen from Fig. 11, which shows that the coated paper had a low WVP when the coating weight was high. When the coating weight was 3.53 g/m^2, WVP was reduced by 10.6%. But when the coating weight was 0.36 g/m^2, WVP decreased by just 4.1%. This

was because, under the equal conditions, the chitosan-coated paper with higher coating weight formed a thicker chitosan film on the surface of the paper, and these films were helpful to hinder water vapor from seeping through the paper.

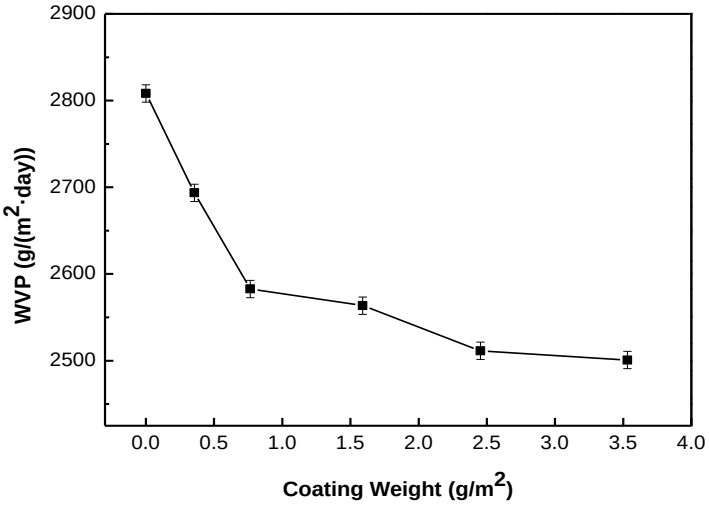


Fig. 11. Effects of coating weight on coated paper WVP

Therefore, the coated paper with higher concentration brought better properties of water vapor barrier. When the stirring speed was 800 rpm, or the dosage ratio of chitosan to acetic acid was 1:0.5, the coated paper had a lower WVP, and had excellent performance as a water vapor barrier. The chitosan-coated paper with a higher coating weight had preferable water vapor barrier properties.

CONCLUSIONS

1. Chitosan was able to form a continuous coating film on the surface of the kraft paper. The good film formation of chitosan on the paper surface could enhance the smoothness. The chitosan solution could not penetrate to the bottom side of kraft paper.
2. The higher concentration of chitosan solution improved its film formation and the smoothness. The film uniformity was the best when the chitosan solution stirring speed was around 800 rpm or the dosage of chitosan was twice that of acetic acid. With the increase of the chitosan coating weight, the smoothness and elongation of the kraft paper all increased.
3. The coated paper had a better effect on resistance to water permeation with a higher chitosan concentration and coating weight. Moreover, when the optimum stirring speed was 800 rpm, or the optimum dosage ratio of chitosan to acetic acid was 1:0.5, the coated paper obtained a preferable effectiveness to resisting water permeation.

4. The optimum process condition on better properties of water vapor barrier were that the chitosan solution had a higher concentration, an optimal pH and stirring speed, as well as higher a coating weight.
5. All in all, chitosan can be used to help to widely develop and apply the paper or board as an environmental kind of packaging material in the packaging industry.

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